

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner  
 US Department of Commerce  
 United States Patent and Trademark  
 Office, PCT  
 2011 South Clark Place Room  
 CP2/5C24  
 Arlington, VA 22202  
 ETATS-UNIS D'AMERIQUE  
 in its capacity as elected Office

Date of mailing (day/month/year) 07 February 2001 (07.02.01)	
International application No. PCT/GB00/02447	Applicant's or agent's file reference CDK 1695
International filing date (day/month/year) 22 June 2000 (22.06.00)	Priority date (day/month/year) 24 June 1999 (24.06.99)
Applicant HAWKINS, John et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:  
 20 December 2000 (20.12.00)

☐ in a notice effecting later election filed with the International Bureau on:  
 \_\_\_\_\_

2. The election ☒ was

☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer R. Chrem Telephone No.: (41-22) 338.83.38
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REC'D 17 JUL 2001

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## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference CDK 1695		<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB00/02447	International filing date (day/month/year) 22/06/2000	Priority date (day/month/year) 24/06/1999	
International Patent Classification (IPC) or national classification and IPC C11D17/00			
Applicant RHODIA CONSUMER SPECIALTIES LIMITED et al.			
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 7 sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of sheets.</p>			
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"><li>I <input checked="" type="checkbox"/> Basis of the report</li><li>II <input type="checkbox"/> Priority</li><li>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</li><li>IV <input checked="" type="checkbox"/> Lack of unity of invention</li><li>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</li><li>VI <input type="checkbox"/> Certain documents cited</li><li>VII <input checked="" type="checkbox"/> Certain defects in the international application</li><li>VIII <input checked="" type="checkbox"/> Certain observations on the international application</li></ul>			
Date of submission of the demand 20/12/2000		Date of completion of this report 13.07.2001	
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465		Authorized officer Ruckebusch, V Telephone No. +49 89 2399 8493 	

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/GB00/02447

**I. Basis of the report**

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

**Description, pages:**

1-14 as originally filed

**Claims, No.:**

1-4 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/02447

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

## IV. Lack of unity of invention

1. In response to the invitation to restrict or pay additional fees the applicant has:

- ☐ restricted the claims.
- ☐ paid additional fees.
- ☐ paid additional fees under protest.
- ☐ neither restricted nor paid additional fees.

2. ☒ This Authority found that the requirement of unity of invention is not complied and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.

3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is

- ☐ complied with.
- ☒ not complied with for the following reasons:  
**see separate sheet**

4. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this report:

- ☒ all parts.
- ☐ the parts relating to claims Nos. .

## V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims 2, 4
	No:	Claims 1, 3
Inventive step (IS)	Yes:	Claims
	No:	Claims 1-4
Industrial applicability (IA)	Yes:	Claims 1-4
	No:	Claims

# **INTERNATIONAL PRELIMINARY EXAMINATION REPORT**

International application No. PCT/GB00/02447

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2. Citations and explanations  
**see separate sheet**

## **VII. Certain defects in the international application**

The following defects in the form or contents of the international application have been noted:  
**see separate sheet**

## **VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:  
**see separate sheet**

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/GB00/02447

- i. Reference is made to the document:

D1: EP-A-0 732 394 (UNILEVER) 18 September 1996

**Re Item IV**

Lack of unity of invention

- iv. Since his invention encompasses various embodiments, the question of unity of the invention might arise (Rule 13.1 PCT) upon entry into the regional phase before the then competent office.

The Applicant has not identified the features which are common to all the embodiments of the invention which should then be claimed and which on their own or in combination represent a contribution over the prior art, and draft the claims accordingly.

**Re Item V**

Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

- v. **Novelty and Inventive Step (Article 33(2) and (3) PCT)**

- v.i The document D1 discloses liquid detergent compositions (see page 3 line 33 to 43, page 4 line 51 to page 5 line 4 and example 13) comprising surfactants, which are an anionic (LAS) and a nonionic (NEODOL <sup>[SPEC0416]</sup> with 9 EO), sorbitol, polymers and builder.

The subject-matter of claims 1 and 3 is therefore not novel, in view of document D1, in the sense of Article 33(2) PCT.

**v.ii** The problem faced by the applicant is:

- the criticality of the amount of deflocculating agent;
- their tendency not to prevent the composition from separating when the temperature varies;
- their tendency to promote foaming;
- forming stable homogeneous compositions.

His solution is to use water soluble carbohydrates; the Applicant found them capable of deflocculating dispersed lamellar or spherulitic surfactant systems; they are also less sensitive to temperature variations.

However, the Applicant:

- compared the **washing performance** of his composition according to example 1 to the washing performance of similar compositions comprising C<sub>12</sub>-C<sub>14</sub> alkyl thiol polyacrylate or alkyl polyglycoside;
- investigated the stability of said composition between 2 and 40°C, which can hardly been said to be extreme temperatures;
- but refrained from investigating whether or not deflocculation had been achieved, for said composition, as for the "comparative" ones.

The Applicant's conclusion as to the capability of a carbohydrate to be a deflocculating agent is not established.

**v.iii** Incidentally, the Examining Authority notes that:

- the Applicant's composition is characterised in that it comprises some deflocculant, said deflocculant itself comprising a carbohydrate ! That definitely falls short saying that a carbohydrate is a deflocculating agent;
- While the composition must be capable of suspending solids, there is no evidence that they actually can;

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/GB00/02447

-the compositions of Examples 3 and 4 are not flocculated, but they both comprise APG and anti-silicone, they thus cannot be relevant when assessing whether or not a carbohydrate is an effective deflocculant agent.

-the composition of Example 1, devoid of thiocyanate and/or the fifty mole ethoxylate is unstable, though comprising a carbohydrate (that confirms that the role of the carbohydrate as a deflocculating agent fulfilling the requisites of the Applicant was not established).

- v.iv** The role of a carbohydrate as a deflocculating agent solving the problem faced by the Applicant can definitely not be acknowledged by the Examining Authority, and the objection of lack of an inventive step in the sense of Article 33(3) PCT is confirmed.

**Re Item VII**

Certain defects in the international application

- vii.** Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the document D1 is not mentioned in the description, nor is this document identified therein.

**Re Item VIII**

Certain observations on the international application

- viii.** The terms "if required", "sufficient" used in claims 1 and 4 are vague and unclear and leave the reader in doubt as to the meaning of the technical features to which they refer, thereby rendering the definition of the subject-matter of said claims unclear (Article 6 PCT).

Claim 1 does not meet the requirements of Article 6 PCT in that the matter for which protection is sought is not clearly defined. The claim attempts to define the subject-matter in terms of the result to be achieved ("structured" is nothing but the expected result) which merely amounts to a statement of the underlying problem. The technical features necessary for achieving this result have not been added.



# PCT

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>CDK 1695</b>	<b>FOR FURTHER ACTION</b> see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. <b>PCT/GB 00/ 02447</b>	International filing date (day/month/year) <b>22/06/2000</b>	(Earliest) Priority Date (day/month/year) <b>24/06/1999</b>
Applicant  <b>RHODIA CONSUMER SPECIALTIES LIMITED et al.</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.  
☒ It is also accompanied by a copy of each prior art document cited in this report.

**1. Basis of the report**

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of Invention is lacking** (see Box II).

**4. With regard to the title,**

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

**5. With regard to the abstract,**

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

**6. The figure of the drawings to be published with the abstract is Figure No.**

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☐ None of the figures.

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 00/02447

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 C11D17/00 C11D3/22

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EP0-Internal, PAJ, CHEM ABS Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 732 394 A (UNILEVER) 18 September 1996 (1996-09-18)	1,3
A	claim 10; example 13	4
A	EP 0 623 670 A (ALBRIGHT & WILSON) 9 November 1994 (1994-11-09) cited in the application page 7, line 40 - line 48; claims 13-17	1,4

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

27 September 2000

Date of mailing of the international search report

05/10/2000

Name and mailing address of the ISA

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Saunders, T

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 00/02447

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
EP 0732394	A	18-09-1996	US	5776883 A	07-07-1998
EP 0623670	A	09-11-1994	AU	678572 B	05-06-1997
			AU	6197994 A	10-11-1994
			BG	62141 B	31-03-1999
			BG	98755 A	31-05-1995
			BR	9401051 A	06-12-1994
			CA	2123017 A	08-11-1994
			CN	1100339 A,B	22-03-1995
			CZ	9401130 A	15-03-1995
			EG	20616 A	30-09-1999
			FI	942106 A	08-11-1994
			GB	2279080 A,B	21-12-1994
			HK	1005746 A	22-01-1999
			HU	68705 A	28-07-1995
			IL	109586 A	05-04-1998
			JP	7126696 A	16-05-1995
			NO	941720 A	08-11-1994
			NZ	260488 A	25-06-1996
			SK	53294 A	12-04-1995
			US	6090762 A	18-07-2000
			ZA	9403170 A	17-01-1995
			CN	1098739 A	15-02-1995
			AT	195334 T	15-08-2000
			CA	2138185 A	16-06-1995
			DE	69425476 D	14-09-2000
			EP	0658620 A	21-06-1995
			GB	2288409 A,B	18-10-1995
			NO	944873 A	16-06-1995

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
4 January 2001 (04.01.2001)

PCT

(10) International Publication Number  
**WO 01/00778 A1**

(51) International Patent Classification<sup>7</sup>: C11D 17/00, 3/22

[GB/GB]: 9 Woodhouse Orchard, Belbroughton, Stourbridge, West Midlands DY9 9TQ (GB).

(21) International Application Number: PCT/GB00/02447

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(74) Agent: **BARKER BRETTELL**; 138 Hagley Road, Edgbaston, Birmingham B16 9PW (GB).

(25) Filing Language: English

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(30) Priority Data:  
9914673.0 24 June 1999 (24.06.1999) GB  
9925699.2 30 October 1999 (30.10.1999) GB

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(71) Applicant (*for all designated States except US*): **RHO-DIA CONSUMER SPECIALTIES LIMITED**, trading as **ALBRIGHT & WILSON SURFACTANTS EUROPE** [GB/GB]; 210-222 Hagley Road West, Oldbury, West Midlands B68 0NN (GB).

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **HAWKINS, John** [GB/GB]; 21 Chandler Avenue, Kinver, South Staffordshire DY7 6AG (GB). **HATCHMAN, Kevan** [GB/GB]; 5 Byland Close, Friarscroft, Bromsgrove, Worcestershire B61 7PL (GB). **CLAPPERTON, Richard, Malcolm**

Published:

— With international search report.

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: **STRUCTURED SURFACTANT SYSTEMS**

(57) Abstract: A structured surfactant composition capable of suspending solids comprises a surfactant, water and, optionally, electrolyte in proportions adapted to form a flocculated, dispersed lamellar and/or spherulitic structured surfactant system, and, in addition, sufficient of a deflocculant to inhibit the flocculation of the system wherein the deflocculant is a water soluble carbohydrate such as sucrose or a hydrophilic derivative of a carbohydrate such as alginate.

WO 01/00778 A1

## **STRUCTURED SURFACTANT SYSTEMS**

The present invention relates to the formulation of structured surfactant suspending systems. It is particularly relevant to the formulation of laundry detergents especially those used for industrial and institutional cleaning.

### **STRUCTURED SURFACTANT**

Suspending solids in liquids presents a problem. If the solids differ in density from the liquid they will tend either to sediment or float. Increasing the viscosity of the liquid can retard, but not prevent such separation, and high viscosities are generally undesirable. Colloidal systems, in which the suspended particles are sufficiently small to experience Brownian motion, e.g. less than 1 micron, may be kinetically stable. However the difficulty or undesirability of comminuting some solids to such sizes, and the impossibility of maintaining many of them at this level in the face of crystal growth or agglomeration, limits the use of colloidal suspensions.

Adjusting the density of one phase to match that of the other is usually impracticable. Moreover such systems are almost always temperature-unstable due to differential rates of thermal expansion.

One method of suspension which permits even relatively large particles to be stably suspended is structured surfactant. The term covers systems in which a surfactant mesophase, usually a lamellar or G-phase, alone or more usually interspersed with an aqueous phase, provides a yield stress which is sufficient, when the system is at rest, to immobilise any suspended particles, but which is sufficiently low to allow the system to be poured like a normal liquid. Such systems may display very low apparent viscosities when stirred, pumped or poured and yet be capable of maintaining particles, sometimes of millimetre or larger size, indefinitely in suspension.

Three main types of suspending system have been employed in practice, all involving a G-phase, in which bilayers of surfactant are arranged with the hydrophobic part of the molecule on the interior and the hydrophilic part on the exterior of the bilayer (or vice versa). The bilayers lie side by side, e.g. in a parallel or concentric configuration, sometimes separated by aqueous layers. G-phases (also known as  $L_\alpha$  phases) can usually be identified by their characteristic textures under the polarising microscope and/or by x-ray diffraction, which is often able to detect evidence of lamellar symmetry. Such evidence may comprise first, second and sometimes third order peaks with d-spacing ( $\frac{2\pi}{Q}$  where  $Q$  is the momentum transfer vector) in a simple integral ratio 1:2:3. Other types of symmetry give different ratios, usually non integral.

Most surfactants form a G-phase either at ambient or at some higher temperature when mixed with water in certain specific proportions. However such G-phases cannot usually be used as structured suspending systems. Useful quantities of solid render them unpourable and smaller amounts tend to sediment.

The main types of structured system used in practice are based on dispersed lamellar, spherulitic and expanded lamellar phases. Dispersed lamellar phases are two phase systems in which the surfactant bilayers are arranged as parallel plates to form domains of G-phases which are interspersed with an aqueous phase to form an opaque gel-like system. They are described in EP O 086 614.

Spherulitic phases comprise well defined spheroidal bodies, usually referred to in the art as spherulites, in which surfactant bilayers are arranged as concentric shells. The spherulites usually have a diameter in the range 0.1 to 15 microns and are dispersed in an aqueous phase in the manner of a classical emulsion, but interacting to form a structured system. Spherulitic systems are described in more detail in EP O 151 884.

Many structured surfactant systems are intermediate between dispersed lamellar and spherulitic, involving both types of structure. Usually systems having a more spherulitic character are preferred because they tend to have lower viscosity. A variant on the spherulitic system comprises prolate or rod shaped bodies sometimes referred to as batonets.

A third type of structured surfactant system comprises an expanded G-phase. It differs from the other two types of structure system in being essentially a single phase, and from conventional G-phase in having a wider d-spacing. Conventional G-phases have a d-spacing of about 5 to 7 nanometers. Attempts to suspend solids in such phases results in stiff pastes which are either non-pourable, unstable or both. Expanded G-phases with d-spacing between 8 and 20, e.g. 10 to 15 nanometers, form when the electrolyte is added to aqueous surfactants at concentrations just below those required to form a normal G-phase, particularly to surfactants in the M phase. The M phase comprises surfactant molecules arranged to form cylindrical rods of indefinite length. It exhibits hexagonal symmetry and a distinctive texture under the polarising microscope. Typical M phases have so high a viscosity that they appear to be curdy solids. M phases near the lower concentration limit (the  $L_1$ /M phase boundary) may be pourable but have a very high viscosity and often a mucous-like appearance. Such systems tend to form expanded G-phases particularly readily on addition of sufficient electrolyte.

Expanded G-phases are described in more detail in EP O 530 708. In the absence of suspended matter they are translucent, unlike dispersed lamellar or spherulitic phases which are necessarily opaque. They are optically anisotropic and have shear dependent viscosity. In this they differ from  $L_1$  phases which are micellar solutions and which include microemulsions.  $L_1$  phases are clear, optically isotropic and substantially Newtonian. They are unstructured and cannot suspend solids. Some  $L_1$  phases exhibit small angle x-ray diffraction spectra which show evidence of hexagonal symmetry. Such phases usually have concentrations near the  $L_1$ /M phase boundary and may form expanded G-phases on addition of electrolyte.

Most structured surfactant systems require the presence of electrolyte as well as surfactant and water in order to form structured systems capable of suspending solids. However certain relatively hydrophobic surfactants such as isopropylamine alkyl benzene sulphonate can form spherulites in water in the absence of electrolyte. Such surfactants are capable of suspending solids in the absence of electrolyte as described in EP O 414 549.

### APPLICATION

Structured surfactants have been applied to the problems of suspending: water insoluble or sparingly soluble builders in laundry detergent; antifoams and enzymes in laundry detergents and other surfactant systems; abrasives in hard surface cleaners; pesticides and oils in agrochemical preparations (EP O 388 239 and EP O 498 231); rock cuttings in drilling muds (EP O 430 602); dyestuffs in dye bath concentrates and printing inks (EP O 472 089); talcs, oils and other cosmetic ingredients in personal care formulations (EP O 530 708).

### FLOCCULATION

A problem with the two phase structured surfactant systems, and especially spherulitic systems, is flocculation of the dispersed surfactant structures. This tends to occur at high surfactant and/or high electrolyte concentration. It can have the effect of making the composition very viscous and/or unstable with the dispersed surfactant separating from the aqueous phase.

Certain amphiphilic polymers have been found to act as deflocculants of structured surfactants. One type of deflocculant polymer exhibits cteniform (comb-shaped) architecture with a hydrophilic backbone and hydrophobic side chains or vice versa. A typical example is a random copolymer of acrylic acid and a fatty alkyl acrylate. Cteniform deflocculants have been described in a large number of patents, for example WO-A-9106622.



A more effective type of deflocculant has surfactant rather than cteniform architecture. With a hydrophilic polymer group attached at one end to a hydrophobic group. Such deflocculants are typically telomers formed by telomerising a hydrophilic monomer with a telogen having a hydrophobic group. Examples of surfactant deflocculants include alkyl thiol polyacrylates and alkyl polyglycosides. Surfactant deflocculants are described in more details in EP O 623 670.

### **THE PROBLEM**

A disadvantage of both surfactant and cteniform deflocculants is that the concentration required to deflocculate to optimum viscosity is critical within fairly narrow limits and varies with temperature. Either too little or too much deflocculant causes instability and/or excessive viscosity. As a result the deflocculated systems tend to separate if the temperature varies significantly.

One approach to the problem of temperature stability has been to add highly cross linked polyacrylates (see US 5 602 092). These are difficult to disperse in the structured liquid.

Another problem is the high cost of the deflocculants and their tendency to promote foaming, which often requires the addition of antifoams.

A particular problem arises in relation to liquid detergents suitable for use in industrial and institutional laundries, such as factories, hospitals and hotels and especially in automatically dosed washing machines.

An ideal laundry detergent for institutional use would combine: high surfactant levels and in particular high levels of non-ionic surfactant, which has been found particularly effective for removing soil; high alkalinity, to saponify fatty soil; and high levels of builder, which improve the performance of the surfactant by counteracting the effects of calcium in the water. The composition must be

homogeneous and pourable and the concentration as high as possible. Unfortunately it is generally difficult to combine surfactants with electrolytes at high concentrations to form stable compositions. It has proved particularly difficult to achieve this with non-ionic surfactants which are not capable of forming stable solutions at high alkalinity or in the presence of electrolyte, except at very low concentrations which are too low to be commercially acceptable. As a consequence it has hitherto been customary to use two separate solutions in institutional machines, one to supply the surfactant and a separate solution as the source of the alkali.

Attempts to combine the two in a single formulation have hitherto been unsuccessful. Even the use of a deflocculant such as those described in EP-A-0 623 670 or EP-A-0 346 995 has not been successful in forming a sufficiently stable homogeneous phase of commercially acceptable concentration, or has done so only over a very restricted temperature range.

### **THE SOLUTION**

We have now discovered that water soluble carbohydrates are capable of deflocculating dispersed lamellar or spherulitic structured surfactant systems. They are substantially cheaper than known deflocculants and do not cause excessive foaming. They are generally less sensitive to temperature variations.

### **THE INVENTION**

Our invention provides a structured surfactant composition capable of suspending solids which comprises surfactant, water and, if required, electrolyte in relative proportions adapted to form a flocculated, dispersed lamellar and/or spherulitic structured surfactant system, and, in addition, sufficient of a deflocculant to inhibit the flocculation of said system characterised in that said deflocculant comprises a water-soluble carbohydrate.

Preferably said surfactant consists of a major amount of non-ionic surfactant and optionally a minor amount of anionic and/or amphoteric. Said water is preferably present in a proportion of from 20 to 60%, especially 30 to 50%. Said electrolyte preferably comprises alkali. Said deflocculant preferably comprises at least 10% by weight of the composition of a mono- or preferably di-saccharide.

### **THE AUXILIARY STABILISER**

The composition may contain an auxiliary stabiliser to prevent or inhibit temperature instability such as bottom separation or phase changes on warming. The stabiliser may comprise (i) an ethoxylated C<sub>8-20</sub>, straight or branched chain alcohol or fatty acid, fatty amine, sorbitan or glycerol ester, alkyl polypropoxy group or alkyl phenyl group and (ii) a water soluble thiocyanate. The number of ethoxy groups may be from 20 to 100, e.g. 30 to 80, preferably 40 to 60. The mole ratio of (i):(ii) may preferably be from 1:100 to 10:1, e.g. 1:50 to 5:1.

The total concentration of auxiliary stabiliser may typically be from 5 to 150% molar based on the deflocculant, e.g. 10 to 100%. The concentration of (B) in the composition is preferably from 0.1 to 10%, e.g. 0.5 to 5%, especially 0.8% to 2%.

### **THE CARBOHYDRATE**

The term "carbohydrate" as used herein includes mono-, di-, and oligosaccharides, water soluble polysaccharides and also water soluble non-surfactant derivatives of the saccharides such as carboxylic acids, e.g. gluconic, mannitol and ascorbic acids and alginates, reduced sugars such as sorbitol, mannitol or inositol and dialdehyde starches.

Preferred deflocculants are sorbitol, alginates, soluble starches and mono and di-saccharides. The alginates are preferably alkali metal or ammonium salts e.g. sodium alginate. They are preferably present in concentrations of from 0.05 to 5%, e.g. 0.1 to 2% especially 0.2 to 1%.

The mono and di-saccharides and derivatives such as sorbitol are usually required in much higher proportions than polysaccharide deflocculants. Generally concentrations of more than 5% by weight of the composition are required, more usually 10 to 20%, especially 12 to 18%.

The di-saccharide is preferably sucrose but may also be, for example, maltose, lactose or cellobiose. Other carbohydrates for use in the invention include glucose, fructose, mannose, ribose, galactose, alldose, talose, gulose, idose, arabinose, xylose, lyxose, erythrose, threose, acrose and rhamiose.

### **INSTITUTIONAL AND INDUSTRIAL FORMULATIONS**

According to a preferred embodiment the invention provides a detergent composition comprising:-

- (A) 20 to 75% by weight of the composition of water;
- (B) At least 3% based on the weight of the composition, preferably 4 to 10%, surfactant comprising more than 50% based on the total weight of surfactant of non-ionic surfactants having a mean HLB of from 10 to 15 and optionally a smaller proportion of anionic and/or amphoteric surfactant;
- (C) At least 10% by weight based on the weight of the composition of builders;
- (D) At least 7% based on the weight of the composition of dissolved non-micelle-forming salts and bases which dissociate at least partially in solution into ions, including any dissolved portion of said builder;
- (E) A total free alkalinity of at least 0.5 normal;

- (F) Sufficient of a deflocculant to provide, in conjunction with components A to E above a pourable composition which does not separate after 1 month at 25°C;

Wherein said deflocculant comprises a water soluble carbohydrate.

The amount of water is typically greater than 30%, preferably greater than 40%, especially greater than 50% and usually less than 70% based on the total weight of the composition.

The surfactant is preferably all non-ionic since in some applications inclusion of anionic surfactant can adversely affect performance. However where anionic surfactant can be tolerated its inclusion has the advantage of enabling higher total concentrations of surfactant to be more easily achieved. Typically wholly non-ionic based formulations contain from 7 to 30%, more typically 10 to 25% by weight surfactant while compositions containing a minor proportion of anionic surfactant may contain up to 50% by weight, e.g. 15 to 40%, especially 20 to 35%.

The non-ionic surfactant preferably consists of from 60 to 100% by weight of alkoxyate, preferably ethoxyate or mixed ethoxyate/propoxyate. Typically it comprises alkoxyated C<sub>8-20</sub>, especially C<sub>10-18</sub> natural or synthetic alcohols. The alcohols are typically primary or secondary, straight or branched chain, saturated or unsaturated. Also effective are alkoxyated fatty acids, fatty amines, alkyl phenols, glyceryl mono and dialkyl esters and sorbitan esters.

The ethoxyate typically contains an average of from 1 to 10 alkoxy groups depending on the alkyl chain length, to give an HLB of from 10 to 15, preferably 12 to 14.

The non-ionic surfactant may comprise a mono- or di- ethanolamide or an amine oxide. The surfactant may optionally contain a minor proportion (i.e. less than 50% based on the total weight of surfactant) of anionic surfactant such as soap and/or

alkyl benzene sulphonate. Other anionic surfactants which may be used include alkyl ether sulphates, alkyl sulphates, olefin sulphonates, paraffin sulphonates and alkyl phosphates.

The builder is preferably sodium tripolyphosphate, but may alternatively be or comprise sodium or potassium pyrophosphate, sodium or potassium citrate, sodium or potassium carbonate or a zeolite. Other builders include EDTA, nitrilotriacetate, phosphonates and poly electrolytes such as polyacrylates or polymaleates. The term "builder", as used herein, excludes any hydroxides used to provide the free alkali but includes carbonate and silicate. The builder is present in amounts greater than 10% by weight based on the total weight of the composition, preferably more than 15%. Levels of builder may be above 20%, any excess over the solubility in the system being present as suspended particles. Builder concentrations do not normally exceed 50% by weight and are usually less than 40%, e.g. less than 30%.

The composition contains a total of at least 7% by weight of dissolved surfactant desolubilising salts and bases. This includes any dissolved portion of the builder and any alkali required to provide the free alkalinity.

It excludes micelle forming components such as an anionic surfactant. The dissolved salts and bases preferably constitute from 10 to 40%, e.g. 15 to 30% by weight of the composition, and sufficient to form a multiphase system in which an aqueous phase is interspersible with a surfactant or surfactant mesophase.

The total free alkali should be sufficient to neutralise at least an equal volume of 0.5 normal HCl. Preferably the free alkalinity is from 0.7 to 2 normal, e.g. 0.8 to 1.5.

The alkyl polyglycoside is preferably a polyglucoside and typically has an average degree of polymerisation between 1.3 and 10, more usually 1.5 to 3. The alkyl polyglycoside is generally added in an amount sufficient to provide an interspersion of the aqueous phase with the surfactant phase at 25°C, which does not separate within 1 month. This may typically require from 0.5 to 10, more usually 1 to 5%, e.g.

2 to 4.5% by weight based on the weight of the composition. The amount is preferably adjusted to obtain a spherulitic composition comprising surfactant vesicles, usually having a multilamellar or G-phase structure, dispersed in an aqueous phase.

We particularly prefer that compositions of the invention contain an auxiliary stabiliser which helps to extend the range of temperature within which the composition is stable. This may be particularly desirable where storage at relatively high temperatures, e.g. 40°C, is an important consideration. The auxiliary stabiliser may be an ethoxylated alcohol having an average of 20 to 100, more usually 25 to 75, e.g. 30 to 60 ethylene oxide groups per molecule, together with a thiocyanate especially potassium thiocyanate. Alternatively the auxiliary stabiliser may be a polycarboxylate having on or more alkyl groups such as C<sub>8-20</sub> alkyl thiol polycarboxylate e.g. polyacrylate or polymaleate, or a copolymer of unsaturated carboxylic acid with a C<sub>8-20</sub> alkyl ester of unsaturated carboxylic acid e.g. a copolymer of acrylic acid and/or maleic acid with a minor proportion of a C<sub>8-20</sub> alkyl acrylate and/or alkyl maleate ester.

The auxiliary stabiliser may be present in proportions up to 5% by weight, usually 0.01 to 3%, e.g. 0.02 to 2 especially 0.01 to 1. Combinations of two or more auxiliary stabilisers may sometimes be particularly effective.

The detergent compositions of the invention preferably also contain the conventional minor detergent ingredients including antifoams such as silicone antifoam, soil suspending agents such as a carboxymethyl cellulose, optical brighteners, stain removers such as enzymes, bleaches including perborate metaborate mixtures, sequestrants such as phosphonates and especially amino phosphonates including aminotris(methylene) phosphonate, ethylene diamine tetrakis (methylene phosphonate), diethylene triamine pentakis (methylene phosphonate) and others in the same series, perfumes, colouring, preservatives, corrosion inhibitors, bleach activators such as TAED and/or fabric conditioner.

The aforesaid minor ingredients may all be present in conventional amounts and usually constitute a total of less than 5% by weight of the composition, typically less than 1%. The anionic component of the ionic ingredients may typically be sodium, potassium or a mixture of the two. Potassium is preferred where very high solids contents are desired.

The invention is illustrated by the following examples in which all proportions are by weight of the 100% material based on the weight of the composition.

#### EXAMPLE 1

	% wt/wt
Optical brightener "TINOPAL" ® CBS/X	0.05
C <sub>12-14</sub> alkyl two mole ethoxylate	3.75
C <sub>12-14</sub> alkyl seven mole ethoxylate	3.75
Potassium thiocyanate	0.5
C <sub>12-18</sub> fifty mole ethoxylate	0.5
Sodium alginate	0.5
Sodium tripolyphosphate	22.5
Formaldehyde	0.05
Water	balance

The above laundry detergent formulation was stable over extended storage periods at ambient, elevated (40°), low (2°) and cycled temperatures and readily pourable. The washing performance was similar to those obtained using either C<sub>12-14</sub> alkyl thiol polyacrylate or alkyl polyglycoside as the deflocculant however use of either of the two latter resulted in excessive foaming, necessitating the addition of an antifoam. The thiol polyacrylate required slightly higher concentrations, and the polyglycoside much higher concentrations, to achieve equivalent viscosity and stability.



When the thiocyanate and/or the fifty mole ethoxylate were omitted from the formulation or from either of the comparative formulations, the resulting products were unstable when stored under fluctuating or elevated temperature conditions.

**EXAMPLE 2**

	% wt/wt
Optical brightener "TINOPAL" ® CBS/X	0.05
C <sub>12-14</sub> alkyl two mole ethoxylate	3.75
C <sub>12-14</sub> alkyl seven mole ethoxylate	3.75
Potassium thiocyanate	0.5
C <sub>12-18</sub> alkyl fifty mole ethoxylate	0.5
Sucrose	15.0
Sodium tripolyphosphate	22.5
Formaldehyde	0.05
Water	balance

The above laundry detergent formulation was stable when stored at ambient, elevated (40°), depressed (2°) and cycled temperatures, was readily pourable and gave washing performance comparable to that of Example 1. Excessive foaming was not observed.

**EXAMPLE 3 AND 4**

The following laundry detergent compositions are stable, pourable, non -flocculated suspensions.

	<b><u>Ex 3</u></b>	<b><u>Ex 4</u></b>
C <sub>12-14</sub> alkyl 6 mole ethoxylate	11.5	11.5
C <sub>12-15</sub> branched alkyl 3 mole ethoxylate	1.0	1.0
C <sub>12-14</sub> alkyl polyglucoside (d.p. 1.6)	2.8	2.7
sodium tripolyphosphate	22.0	22.0
sodium hydroxide	7.0	7.0
sodium silicate	1.0	1.0
sorbitol	15.0	2.0
optical brightner "TINOPAL"® cbs/x	0.1	0.1
polyvinyl pyrrolidone	-	0.05
silicone antifoam	0.1	0.1
sodium triethylenetetramine		
pentapethylen phosphonate	0.25	-
Conductivity	15.2mS	31.6mS
Viscosity	4000cp	2000cp
Sisko Index	0.59	0.4

**CLAIMS**

1. A structured surfactant composition capable of suspending solids which comprises surfactant, water and, if required, electrolyte in relative proportions adapted to form a flocculated, dispersed lamellar and/or spherulitic structured surfactant system, and, in addition, sufficient of a deflocculant to inhibit the flocculation of said system characterised in that said deflocculant comprises a water-soluble carbohydrate.
2. A composition according to claim 1 where said carbohydrate is an alginate present in a proportion of from 0.05% to 5% by weight based on the weight of the composition.
3. A composition according to claim 1 wherein said carbohydrate is a mono or disaccharide or derivative thereof in a proportion of from 10 to 20% by weight based on the weight of the composition.
4. A detergent composition comprising :-
  - (A) 20 to 75% by weight of the composition of water;
  - (B) At least 3% based on the weight of the composition, preferably 4 to 10%, surfactant comprising more than 50% based on the total weight of surfactant of non-ionic surfactants having a mean HLB of from 10 to 15 and optionally a smaller proportion of anionic and/or amphoteric surfactant;
  - (C) At least 10% by weight based on the weight of the composition of builders;
  - (D) At least 7% based on the weight of the composition of dissolved non-micelle-forming salts and bases which dissociate at least partially in solution into ions, including any dissolved portion of said builder;

- (E) A total free alkalinity of at least 0.5 normal;
- (F) Sufficient of a deflocculant to provide, in conjunction with components A to E above a pourable composition which does not separate after 1 month at 25°C;

Wherein said deflocculant comprises a water soluble carbohydrate.

# INTERNATIONAL SEARCH REPORT

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## A. CLASSIFICATION OF SUBJECT MATTER

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## B. FIELDS SEARCHED

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 732 394 A (UNILEVER) 18 September 1996 (1996-09-18)	1, 3
A	claim 10; example 13 ----	4
A	EP 0 623 670 A (ALBRIGHT & WILSON) 9 November 1994 (1994-11-09) cited in the application page 7, line 40 - line 48; claims 13-17 -----	1, 4

☐ Further documents are listed in the continuation of box C.



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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
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(54) Title: STRUCTURED SURFACTANT SYSTEMS

(57) Abstract: A structured surfactant composition capable of suspending solids comprises a surfactant, water and, optionally, electrolyte in proportions adapted to form a flocculated, dispersed lamellar and/or spherulitic structured surfactant system, and, in addition, sufficient of a deflocculant to inhibit the flocculation of the system wherein the deflocculant is a water soluble carbohydrate such as sucrose or a hydrophilic derivative of a carbohydrate such as alginate.

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